

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 4

LIQUID INDUSTRIAL WASTE SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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G. C. RONAN, DIRECTOR
Laboratory Services Branch
Ministry of the Environment

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1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 4

LIQUID INDUSTRIAL WASTE SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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Laboratory Services Branch
Ministry of the Environment

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ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT 1986

INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robin. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

sd = **sqrt[{{(sumx2 - (sumx)^2)/n}/(n-1)}]**I
sd = **sqrt(sumd2/2n)**II

where : x = the individual values; n = the number of events
 d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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4. Industrial Wastes

Industrial waste samples are highly variable in source and composition making these sample types the most difficult to analyse and maintain quality assurance.

Table 4.1 summarizes the parameters determined, the preparation methods used and the instrument types used for the analysis industrial wastes.

TABLE 4.1

Parameter	Collection Device	Preparation	Analysis
Metals	Plastic or glass jars	Acid Digest	ICP - AES
Anions	Plastic or glass jars	Filtration	IC
Cyanide	Plastic or glass jars	Distillation	Colorimetry
Sulfide	Plastic or glass jars		Colorimetry

4.1 Industrial Waste Quality Assurance

Sub aliquots of industrial waste samples are analysed separately to generate duplicate results. Blanks consist of the digestion acid or distilled water as appropriate.

Industrial waste samples frequently have solid, aqueous and organic liquid phases. In general, only the aqueous phase is sampled for inorganic analyses, and the QA materials reflect this position. Composite solutions are spiked to achieve measurable levels of parameters determined.

Table 4.2 summarizes the QA materials used when industrial waste samples are being analysed.

TABLE 4.2

Sample Designation	Type	Parameter
qcal,qcbl	Standard solution	Cyanide
qcd	Standard solution (0.2 mg/L)	Cyanide
QCIW-1	Composite landfill spiked	ICP Metals
475-3	EPA solution	Hydride Metals

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE:TE-Ind. Waste
UNIT:Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.08 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.03 to 5.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.944 mg/L	2.40
std. dev.	.843 mg/L	0.36
R.S.D.	89.3 %	15.3 %

Precision of Duplicates-low range mid range high range

s.d.	.051	0.14	0.38
------	------	------	------

mean	.371	1.51	3.46
------	------	------	------

W .1 mg/L T .5 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA
ALUMINUM IN INDUSTRIAL WASTE

Operating Range = 0.030 to 5.0 mg/L

IN - RUN DUPLICATES

Range	<0.030	0.030 to 1.00	1.00 to 2.50	2.50 to 5.0	>5.0
no.	24	16	8	5	3
s.w.		0.0506	0.1420	0.3758	
mean		0.3709	1.5126	3.4626	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	7	0.944	0.8425	89.25
QCLF-3	59	2.339	0.3568	15.25

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	66	0.478	0.8550

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ARSENIC
UNIT: Biomaterials

TEST CODE: ASUT SAMPLE TYPE: TE-Ind. Waste
SUPERVISOR: R. Sadana

METHOD CODE: 510CF3

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Glass bottle with bakelite screw cap (16 oz.)

Preservative- 1ml conc. HNO₃ for sample filling 16 oz. bottle

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Twenty ml of sample is pipetted into 20x150 mm pyrex test tube. A batch of sixty-eight tubes including samples, blanks, standards and controls are run. These samples are fed to an automated system for measurement of selenium by the hydride-FAAS technique.

Samples with selenium concentration exceeding 10 ng/ml are reanalyzed after an acid digestion. They are digested by taking 20 ml sample aliquot in a 100 ml beaker and by adding 4 ml of 6:3:1 HNO₃:HClO₄:H₂SO₄. Heat until dense white fumes. Cool, add .5 ml of H₂O and 2.5 ml of HCl, transfer to test tube calibrated at 20 ml, bring to mark, mix well, and analyze.

INTERFERENCES: Excessive concentrations of Cu, Fe, Ni

REPORTING RESULTS: mg/L-2 dec. if <10, 1 dec. if 10-100, 0 dec. if >100

INSTRUMENTATION: Atomic Absorption Spectrophotometer (Varian 1200) with strip chart recorder, peristaltic pump, auto-sampler, open-ended and heated quartz 'T' atomizer, and gas-liquid separator.

Calibration Range: 0 to 40 ng/ml

Resolution: 0.01 absorbance

Sensitivity: 20 ng/ml reads 0.150 Abs.

Instrument Detection Limit: 1 ng/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.001 to 0.04 mg/L

Accuracy-

Precision of Controls-

	A
mean	.413 mg/L
std. dev.	.011 mg/L
R.S.D.	2.7 %

B

Precision of Duplicates-low range	mid range	high range
s.d. .0002	0.00	
mean .002		.014
W .001 mg/L	T .005 mg/L	

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN INDUSTRIAL WASTE

Operating Range = 0.001 to 0.04 mg/L

IN - RUN DUPLICATES

Range	<0.001	0.001 to 0.01	0.01 to 0.02	0.02 to 0.04	>0.04
no.	3	13	2	0	1
s.w.		0.0002	0.0000	0.0000	
mean		0.0020	0.0140	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
475-3	19	0.413	0.0110	2.66

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BARIUM TEST CODE: BAUT SAMPLE TYPE: TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour
50.0 ml into a calibrated test tube; for highly coloured samples or
those containing larger amounts of amounts of solids, use 5.0 ml. Add
1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-
regia. Make volume to 12.5 ml. Metal concentration to be determined by
Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too
viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and
ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia
and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer,
Jobin-Yvon JY48P equipped with autosampler and DEC computer system for
concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10.0 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.008 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.005 to 2.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.853 mg/L	0.207
std. dev.	.047 mg/L	0.024
R.S.D.	5.6 %	11.8 %

Precision of Duplicates-low range	mid range	high range
s.d. .004	.017	0.57
mean .065	.474	1.41

W .01 mg/L

T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of
the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA
BARIUM IN INDUSTRIAL WASTE

Operating Range = 0.005 to 2.0 mg/L

IN - RUN DUPLICATES

Range	<0.005	0.005 to 0.40	0.40 to 1.00	1.00 to 2.0	>2.0
no.	12	41	1	1	1
s.w.		0.0035	0.0173	0.5728	
mean		0.0650	0.4744	1.4121	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.853	0.0474	5.56
QCLF-3	62	0.207	0.0244	11.78

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	58	0.004	0.0071

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BERYLLIUM TEST CODE: BEUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10.0 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit:

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range mid range high range

s.d.

mean

W .01 mg/L

T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

BERYLLIUM

IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	56	0	0	0	0
s.w.		0.0000	0.0000	0.0000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	0	0.000	0.0000	0.00
QCLF-3	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	28	0.001	0.0009

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CADMIUM TEST CODE: CDUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2
REVISION NO:
NATURE OF LAST REVISION:

DATE:

SAMPLE HANDLING:

Quantity Required- 300 ml
Container- 500 ml plastic or glass
Preservative- HNO₃
Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour
50.0 ml into a calibrated test tube; for highly coloured samples or
those containing larger amounts of amounts of solids, use 5.0 ml. Add
1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-
regia. Make volume to 12.5 ml. Metal concentration to be determined by
Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too
viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and
ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia
and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES:

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer,
Jobin-Yvon JY48P equipped with autosampler and DEC computer system for
concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .002 to 0.100 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.941 mg/L	1.79
std. dev.	.034	0.13
R.S.D.	3.6 %	7.4 %

Precision of Duplicates-low range	mid range	high range
s.d. .0015	.0002	
mean .0056	.0345	

W .002 mg/L

T .010 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of
the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN INDUSTRIAL WASTE

Operating Range = 0.002 to 0.1 mg/L

IN - RUN DUPLICATES

Range	<0.002	0.002 to 0.02	0.02 to 0.05	0.05 to 0.1	>0.1
no.	46	6	2	0	2
s.w.		0.0015	0.0002	0.0000	
mean		0.0056	0.0345	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.941	0.0336	3.57
QCLF-3	62	1.791	0.1327	7.41

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	31	0.002	0.0027

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: .02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.2 to 100 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	19.8 mg/L	296
std. dev.	6.2	21
R.S.D.	31.3 %	7.0 %

Precision of Duplicates-low range	mid range	high range	B
s.d.	0.2	0.9	4.2
mean	10.2	35.6	71.8

W .5 mg/L

T 2.5 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM IN INDUSTRIAL WASTE

Operating Range = 0.200 to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.200	0.200 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	1	7	15	22	11
s.w.	0.2141		0.8772	4.1631	
mean	10.2219		35.6429	71.8201	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	19.798	6.1970	31.30
QCLF-3	62	295.791	20.8304	7.04

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	49	1.506	2.4736

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CHROMIUM TEST CODE: CRUT SAMPLE TYPE: TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.01 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.910 mg/L	1.76
std. dev.	.062 mg/L	0.19
R.S.D.	6.8 %	10.6 %

Precision of Duplicates-low range mid range high range

s.d. .008 .008 .017

mean .039 .384 .518

W .01 mg/L

T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA
CHROMIUM IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	33	11	1	2	9
s.w.		0.0083	0.0077	0.0170	
mean		0.0392	0.3835	0.5176	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.910	0.0615	6.76
QCLF-3	62	1.756	0.1868	10.64

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	50	0.006	0.0086

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COBALT TEST CODE: COUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2
REVISION NO:
NATURE OF LAST REVISION:

DATE:

SAMPLE HANDLING:

Quantity Required- 300 ml
Container- 500 ml plastic or glass
Preservative- HNO₃
Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour
50.0 ml into a calibrated test tube; for highly coloured samples or
those containing larger amounts of amounts of solids, use 5.0 ml. Add
1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-
regia. Make volume to 12.5 ml. Metal concentration to be determined by
Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too
viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and
ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia
and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer,
Jobin-Yvon JY48P equipped with autosampler and DEC computer system for
concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: .02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	1.01 mg/L	1.84
std. dev.	0.36	0.10
R.S.D.	3.6 %	5.3 %

Precision of Duplicates-low range	mid range	high range
s.d.	.004	.008
mean	.053	.351

W .01 mg/L

T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of
the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT

IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	48	6	1	1	0
s.w.		0.0036	0.0075	0.0290	
mean		0.0531	0.3507	0.9250	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	1.005	0.0359	3.58
QCLF-3	62	1.839	0.0981	5.34

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	43	0.002	0.0034

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.006 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.005 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.982 mg/L	1.91
std. dev.	.086 mg/L	0.16
R.S.D.	8.8 %	8.5 %

Precision of Duplicates-low range	mid range	high range
s.d.	0.031	0.114
mean	0.077	0.265

W 0.02 mg/L

T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN INDUSTRIAL WASTE

Operating Range = 0.005 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.005	0.005 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	22	19	3	4	8
s.w.		0.0302	0.1140	0.0119	
mean		0.0765	0.2648	0.7113	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.982	0.0860	8.76
OCLF-3	62	1.911	0.1620	8.48

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.011	0.0203

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: IRON TEST CODE: FEUT SAMPLE TYPE: TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2
REVISION NO:
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml
Container- 500 ml plastic or glass
Preservative- HNO₃
Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.03to 20 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	2.58 mg/L	2.75
std. dev.	0.93 mg/L	1.03
R.S.D.	36.0 %	37.5 %

Precision of Duplicates-low range mid range high range

s.d. .28 0.15 00.26

mean .80 6.80 15.91

W .1 mg/L T .5 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN INDUSTRIAL WASTE

Operating Range = 0.030 to 20.0 mg/L

IN - RUN DUPLICATES

Range	<0.030	0.030 to 4.00	4.00 to 10.00	10.00 to 20.0	>20.0
no.	17	27	2	3	7
s.w.		0.2772	0.1467	0.2620	
mean		0.8001	6.7953	15.9137	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	19	2.581	0.9297	36.02
QCLF-3	50	2.750	1.0322	37.53

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	24	0.020	0.0498

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: LEAD TEST CODE: PBUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2
REVISION NO:
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml
Container- 500 ml plastic or glass
Preservative- HNO₃
Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour
50.0 ml into a calibrated test tube; for highly coloured samples or
those containing larger amounts of amounts of solids, use 5.0 ml. Add
1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-
regia. Make volume to 12.5 ml. Metal concentration to be determined by
Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too
viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and
ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia
and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer,
Jobin-Yvon JY48P equipped with autosampler and DEC computer system for
concentration print-out; Commodore Pet microcomputer interface to LIS.
Calibration Range:0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit:0.02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.03 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.919 mg/L	0.695
std. dev.	.065 mg/L	0.210
R.S.D.	7.0 %	30.2 %

Precision of Duplicates-low range	mid range	high range
s.d. .028	.009	.010
mean .091	.225	.824

W .02 mg/L

T .10 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD

IN INDUSTRIAL WASTE

Operating Range = 0.030 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.030	0.030 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	45	6	1	1	3
s.w.		0.0275	0.0086	0.0096	
mean		0.0912	0.2246	0.8241	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.919	0.0646	7.03
QCLF-3	62	0.695	0.2099	30.21

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.017	0.0303

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MAGNESIUM TEST CODE: MGUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.2 to 100.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	4.67 mg/L	293
std. dev.	0.86 mg/L	24
R.S.D.	18.8 %	8.1 %

Precision of Duplicates-low range mid range high range

s.d. 0.18 0.8

mean 8.27 35.7

W .5 mg/L

T 2.5 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN INDUSTRIAL WASTE

Operating Range = 0.200 to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.200	0.200 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	1	36	18	0	1
s.w.		0.1770	0.8317	0.0000	
mean		8.2736	35.7383	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	4.665	0.8755	18.77
QCLF-3	62	292.842	23.8079	8.13

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	39	0.140	0.3778

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MANGANESE TEST CODE: MNUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.003 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .01 to 2.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.996 mg/L	2.19
std. dev.	.029 mg/L	0.13
R.S.D.	2.9 %	5.9 %

Precision of Duplicates-low range mid range high range

s.d.	.010	.002	0.021
mean	.104	.470	1.654

W .01 mg/L T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN INDUSTRIAL WASTE

Operating Range = 0.010 to 2.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.40	0.40 to 1.00	1.00 to 2.0	>2.0
no.	12	35	2	1	6
s.w.		0.0095	0.0016	0.0205	
mean		0.1038	0.4694	1.6544	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.996	0.0287	2.88
QCLF-3	62	2.190	0.1283	5.86

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	34	0.003	0.0029

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MOLYBDENUM TEST CODE: MOUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer program

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.886 mg/L	0.666
std. dev.	.026 mg/L	0.106
R.S.D.	3.0 %	15.9 %

Precision of Duplicates-low range mid range high range

s.d. .002 .004 .022

mean .049 .324 .819

W .01 mg/L

T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	38	12	1	5	0
s.w.		0.0019	0.0042	0.0220	
mean		0.0494	0.3239	0.8185	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.886	0.0262	2.96
QCLF-3	62	0.666	0.1059	15.92

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.007	0.0126

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NICKEL TEST CODE: NIUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour
50.0 ml into a calibrated test tube; for highly coloured samples or
those containing larger amounts of amounts of solids, use 5.0 ml. Add
1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-
regia. Make volume to 12.5 ml. Metal concentration to be determined by
Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too
viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and
ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia
and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer program.

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer,
Jobin-Yvon JY48P equipped with autosampler and DEC computer system for
concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.887 mg/L	1.75
std. dev.	.059 mg/l	0.13
R.S.D.	6.6 %	7.6 %

Precision of Duplicates-low range	mid range	high range
s.d. .005	.005	.006
mean .044	.266	.603

W 0.01 mg/L

T 0.05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of
the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA
NICKEL IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	32	10	4	3	7
s.w.		0.0054	0.0050	0.0057	
mean		0.0444	0.2656	0.6032	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.887	0.0587	6.61
QCLF-3	62	1.746	0.1320	7.56

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.015	0.0270

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SILVER TEST CODE: AGUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit:

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range mid range high range

s.d. .006

mean .046

W

T

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

SILVER

IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	55	1	0	0	0
s.w.		0.0057	0.0000	0.0000	
mean		0.0460	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	0	0.000	0.0000	0.00
QCLF-3	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	12	0.012	0.0141

DATE 87/03/12

SUMMARY REPORT OF QUALITY CONTROL DATA
SELENIUM IN INDUSTRIAL WASTE

Operating Range = 0.001 to 0.04 mg/L

IN - RUN DUPLICATES

Range	<0.001	0.001 to 0.01	0.01 to 0.02	0.02 to 0.04	>0.04
no.	9	5	0	0	1
s.w.		0.0000	0.0000	0.0000	
mean		0.0010	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
475-3	15	0.072	0.0060	8.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: STRONTIUM TEST CODE: SRUT SAMPLE TYPE:TE-Ind. Waste
UNIT:Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10.0 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit:

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.951 mg/L	5.60
std. dev.	.064 mg/L	0.36
R.S.D.	6.8 %	7.9 %

Precision of Duplicates-low range	mid range	high range
s.d. .004	.011	.017
mean .117	.310	.771

W .01 mg/L

T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

STRONTIUM IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	2	24	14	5	11
s.w.		0.0036	0.0112	0.0167	
mean		0.1170	0.3096	0.7707	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.951	0.0643	6.76
QCLF-3	62	4.598	0.3624	7.88

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	43	0.023	0.0392

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: TITANIUM TEST CODE: TIUT SAMPLE TYPE:TE-Ind. Waste
UNIT: Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml

Container- 500 ml plastic or glass

Preservative- HNO₃

Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit:

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.817 mg/L	0.558
std. dev.	.016 mg/L	0.035
R.S.D.	2.0 %	6.3 %

Precision of Duplicates-low range	mid range	high range
s.d.	.014	.005
mean	.075	.280

W .02 mg/L

T .10 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

TITANIUM

IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	39	15	1	1	0
s.w.		0.0140	0.0052	0.0059	
mean		0.0748	0.2798	0.5336	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.817	0.0163	2.00
QCLF-3	50	0.558	0.0352	6.32

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	51	0.008	0.0240

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: VANADIUM TEST CODE: VVUT SAMPLE TYPE:TE-Ind. Waste
UNIT:Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2
REVISION NO:
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml
Container- 500 ml plastic or glass
Preservative- HNO₃
Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION:Partial Extn.- Total Extn.-100 % Extracted-

Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES: Several, compensated for by computer

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.008 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.809 mg/L	0.580
std. dev.	.035 mg/L	0.102
R.S.D.	4.4 %	17.6 %

Precision of Duplicates-low range mid range

s.d. .005

mean .146

high range

W .01 mg/L T .05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM

IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	53	3	0	0	0
s.w.		0.0050	0.0000	0.0000	
mean		0.1464	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.809	0.0353	4.36
QCLF-3	52	0.580	0.1020	17.60

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	35	0.007	0.0074

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE:TE-Ind. Waste
UNIT:Ind. Dom. & Landfill Waste SUPERVISOR: J. Pimenta

METHOD CODE: 539AE2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 300 ml
Container- 500 ml plastic or glass
Preservative- HNO₃
Other- Refrigerate samples until analyzed.

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Shake sample. For clear samples with little sediment, pour 50.0 ml into a calibrated test tube; for highly coloured samples or those containing larger amounts of amounts of solids, use 5.0 ml. Add 1.0 ml conc. HNO₃ and dry in oven at 105°C. Digest residue with aqua-regia. Make volume to 12.5 ml. Metal concentration to be determined by Atomic Spectroscopy (AAS or ICP-AES).

Oily samples: Pipette 5.0 ml into a Vycor crucible (If sample is too viscous for pipetting weigh 5.0 g). Absorb oil in cellulose powder and ignite in a muffle furnace for 3 hours. Digest the ash with aqua-regia and make to a final volume of 12.5 ml. Analyze by Atomic Spectroscopy.

INTERFERENCES:

REPORTING RESULTS: mg/L (μ g/g for oil) to 2 places after the decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 to 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: .005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.01 to 1.0 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.914 mg/L	1.69
std. dev.	.042	0.25
R.S.D.	4.6 %	15.1 %

Precision of Duplicates-low range mid range high range

s.d.	.010	.025	.106
------	------	------	------

mean	.057	.319	.781
------	------	------	------

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS: Analysis repeated if value for control exceeds $\pm 15\%$ of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC

IN INDUSTRIAL WASTE

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	11	24	5	4	12
s.w.		0.0097	0.0246	0.1059	
mean		0.0566	0.3188	0.7808	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCIW-1	26	0.914	0.0419	4.59
QCLF-3	62	1.689	0.2541	15.05

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	66	0.032	0.0776

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total cyanide TEST CODE: CCNAUR SAMPLE TYPE:IW-Liquid(TE)
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:001AC2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml

Container- Glass or plastic (preferred)

Preservative- NaOH

Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-100

Procedure- The sample is first run to see if there is cyanide present. The is run directly by the automated high temperature distillation with 25% H₃PO₄-5% H₃PO₂ followed by a colourimetric analysis with chloramine T -isonicotinic acid -barbituric acid method.

If the total cyanide is > .01 mg/L then 5 to 250 ml of sample is manually distilled with 30 ml of 15%(w/v)tartaric acid. The distillate is collected in 50 ml of 1N NaOH, and analyzed by the automated Technicon distillation system referred to above.

INTERFERENCES: SCN interference is removed by distillation.

Distillable organics may interfere; also S= at high levels.

REPORTING RESULTS:Mg/l CN: 3 decimal places up to 3 significant figs

INSTRUMENTATION: Technicon AAII continuous flow analyzer

including pump, colourimeter, appropriate autosampler and recorder.

High temperature distillation apparatus (Technicon). Manual dist. app

Calibration Range: 0 to 0.2 mg/l as CN

Resolution: 0.001 mg/l

Sensitivity:

Instrument Detection Limit: 0.001 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.00100 to 0.200 mg/l

Accuracy- 100%

Precision of Controls-

	A	B
mean	.110 mg/L	0.059
std. dev.	.0027mg/L	0.0026
R.S.D.	2.45 %	4.41 %

Precision of Duplicates-low range mid range high range

s.d.	0.0011	0.0012
mean	0.034	0.132

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS: Pure CN standards are recovered 100% during manual distillation. Complex cyanides can normally be expected to be recovered at 100%.

SUMMARY REPORT OF QUALITY CONTROL DATA
TOTAL CYANIDE IN INDUSTRIAL WASTE

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

Range	<0.001	0.001 to 0.08	0.08 to 0.2	0.2 to 0.4	>0.4
no.	0	12	1	0	3
s.w.		0.0004	0.0141	0	
mean		0.0069	0.16	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	146	0.149	0.0049	3.29
qc-b	146	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	146	0.001	0

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Free cyanide TEST CODE: CCNFUR SAMPLE TYPE:IW-liquid(TE)
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:700AC2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Glass or plastic (preferred)

Preservative- NaOH

Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted- *

Procedure- Pass sample aliquot through an automated low temperature distillation (106°C) in a distillation acid consisting of 10% acetic acid and 0.5% zinc acetate.

Analyze distillate by the Chloramine-T -pyridine-barbituric acid colourimetric method, or equivalent.

INTERFERENCES: None

REPORTING RESULTS:Mg/l CN to 2 decimal places up to 3 significant figs

INSTRUMENTATION: Technicon automated continuous flow analyzer including pump, colourimetric distillation apparatus and sampler; suitable recorder.

Calibration Range: 0 to 0.2 mg/l as CN

Resolution: 0.001

Sensitivity:

Instrument Detection Limit: 0.001 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0100 to 0.400 mg/l

Accuracy- 100%

Precision of Controls-

	A	B
mean	.110 mg/L	0.060
std. dev.	.0036mg/L	0.0031
R.S.D.	3.27 %	5.17%

Precision of Duplicates-low range mid range high range

s.d. 0.0026

mean 0.0202

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS: * The test defines the results reported in this case. The terminology "Weak Acid Dissociable" is commonly used and represents weakly associated cyanide compounds such as KCN, NaCN, NiCN₄, HCN, etc.

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CYANIDE IN INDUSTRIAL WASTE

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

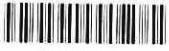
Range	<0.001	0.001 to 0.08	0.08 to 0.2	0.2 to 0.4	>0.4
no.	1	9	2	1	1
s.w.		0.0002	0.001	0.0028	
mean		0.0037	0.176	0.358	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	135	0.151	0.0062	4.11
qc-b	135	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	135	0.001	0



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